

PHOSPHORUS RECOVERY FROM POULTRY LITTER

A. A. Szögi, M. B. Vanotti, P. G. Hunt

ABSTRACT. Land application of large amounts of poultry litter is an environmental concern often associated to excess phosphorus (P) in soils and potential pollution of water resources. Recovery of P from poultry litter waste is an attractive approach when on-farm application of poultry litter is not an option. A treatment process, called “quick wash,” was developed for extraction and recovery of P from poultry litter and animal manure solids. The quick wash process consists of three consecutive steps: (1) P extraction, (2) P recovery, and (3) P recovery enhancement. In step 1, organically bound P is converted to soluble P by rapid hydrolysis reactions using selected mineral or organic acids. This step also releases P from insoluble inorganic phosphate complexes. The washed litter residue is subsequently separated from the liquid extract and dewatered; unnecessary carbon (C) and nitrogen (N) transformations are prevented by dewatering the residue. In step 2, P is precipitated by addition of lime to the liquid extract to form an alkaline earth metal-containing P product. In step 3, an organic poly-electrolyte is added to enhance the P grade of the product. This approach of extracting and recovering P from poultry litter using the quick wash process produces a final P product that can be reused as fertilizer. In addition, the remaining washed solid residue has a more balanced N to P (N:P) ratio that is more environmentally safe for land application and use by crops. As an alternative, washed poultry litter residue can be used for energy production or reutilized as bedding, especially in areas where bedding material is in short supply.

Keywords. Animal waste treatment, CAFO, Fertilizer, Phosphorus recovery, Phosphorus recycling, Poultry litter.

Phosphorus (P) buildup to elevated levels in crop soils due to intense land application of animal manures has the potential to wash out as field runoff and pollute nearby surface waters (Edwards and Daniel, 1992; Heathwaite et al., 2000; Sharpley et al., 2000, 2007). This is a national concern affecting dairy, swine, and poultry production systems when land is limiting. To solve accumulation and distribution problems of this nutrient, a substantial amount of manure P may require to be moved at least off the farm or longer distances beyond county limits (USDA-ERS, 2000). Thus, the ability to remove P from manure will be critical to livestock and poultry producers to accomplish manure utilization through land application without elevating soil P levels. In addition, the aspect of P reuse is becoming important for the fertilizer industry because the world P reserves are limited (Smil, 2000). On one hand, the U.S. annual consumption of inorganic P for crop production is about 1680 million kg (Potash and Phosphate Institute, 2002). On the other hand, for the U.S. as a whole, confined livestock produces 650 million kg of recoverable manure P annually, with 65% (420 million kg) in excess of

on-farm needs (Kellogg et al., 2000). Therefore, reuse of P recovered from animal waste could substitute about 25% of the P now obtained from mining. Given that a major portion of the recoverable manure P (250 million kg) is supplied by poultry manure (Kellogg et al., 2000), new manure P management technologies are needed for environmentally sustainable poultry production (Sharpley et al., 2007).

State-of-the-art manure management technologies and programs available to solve the problem of P in poultry operations include: (1) improved manure application methods, such as immobilization of P with alum to prevent runoff (Moore, 2002; Sharpley et al., 2007); (2) energy generation by combustion (USDOE, 2000), gasification (Sheth and Turner, 2002), or anaerobic digestion (Kelleher et al., 2002); and (3) transport of waste or compost to agricultural lands with low levels of P (Jones and D’Souza, 2001; Kelleher et al., 2002; Keplinger and Hauck, 2004). As an alternative, we have invented a process (“quick wash”) consisting of a rapid extraction and subsequent recovery of P in solid form from solid manure prior to land application (Szögi et al., 2008). Our approach has three distinctive advantages over the state of the art: (1) compared with alum immobilization that binds P in a form not useful for plant use, the recovered P from the quick wash produces a valuable product that can be reused as fertilizer; (2) compared to co-firing and gasification processes, the residual organic matter is conserved for additional soil benefit; and (3) compared with solid manure transport programs, there is no need to transport large volumes of manure, since only about 15% of the initial volume leaves the farm containing the concentrated P product. The remaining solid residue (washed manure or litter) has a more balanced nitrogen:phosphorus (N:P) ratio that is more environmentally safe for land application and use by crops. In this article, we describe the development and technical feasibility of a new treatment

Submitted for review in December 2007 as manuscript number SE 7311; approved for publication by the Structures & Environment Division of ASABE in September 2008.

Mention of trade names or commercial products in this article is solely for the purpose of providing specific information and does not imply recommendation or endorsement by the USDA.

The authors are **Ariel A. Szögi, ASABE Member**, Soil Scientist, **Matias B. Vanotti, ASABE Member Engineer**, Soil Scientist, and **Patrick G. Hunt, ASABE Member**, Soil Scientist, USDA-ARS Coastal Plains Soil, Water, and Plant Research Center, Florence, South Carolina. **Corresponding author:** Ariel A. Szögi, USDA-ARS Coastal Plains Soil, Water, and Plant Research Center, 2611 West Lucas St., Florence, SC 29501; phone: 843-669-5203, ext. 109; fax: 843-669-6970; e-mail: Ariel.Szogi@ars.usda.gov.

process leading to extraction and recovery of P from poultry litter.

MATERIALS AND METHODS

PROCESS CONFIGURATION

The quick wash process consists of three consecutive steps: (1) P extraction, (2) P recovery, and (3) P recovery enhancement. In the first step, poultry litter is washed by mixing it with water and acid in a reactor vessel at pH lower than 5.0 (fig. 1). The washed litter residue is further settled and dewatered to prevent unnecessary carbon (C) and nitrogen (N) oxidation and digestion. This first step produces a liquid extract containing low suspended solids ($<3 \text{ g L}^{-1}$) and extracted soluble P. The liquid extract is transferred to a second vessel where P is recovered (steps 2 and 3, fig. 1).

In the step 1 of the process (fig. 1), organically bound P is converted to soluble P by rapid hydrolysis reaction with the acid solution, and this rapid hydrolysis reaction has the capacity to extract large amounts of P from poultry litter. This step also releases P from insoluble inorganic phosphate complexes. The hydrolysis and solubilization of P compounds are obtained by using organic acids (such as citric, oxalic, malic, etc.), mineral acids (such as hydrochloric or sulfuric), or a mixture of both mineral and organic acids.

In step 2, P in the liquid extract is precipitated by addition of an alkaline earth base to a pH range of 9.0 to 11.0 to form alkaline earth metal-containing P compounds. Subsequently in step 3, an organic flocculant is added into the second vessel to enhance precipitation and P grade of the precipitated product (fig. 1). After a settling period, the precipitated P-rich solid is removed from the bottom of the second vessel while the supernatant liquid is recycled back into the quick wash system or land applied. Steps 1 to 3 can be carried out in batch mode using for example a single vessel to do the chemical mixing and settling, or adapted for continuous operation using two separate vessels to do the mixing first and then the settling, as shown in figure 1.

Three experiments were done to test the quick wash process. The first experiment tested organic and inorganic acids for their potential to extract P from the solids (step 1). The second experiment was done to demonstrate the removal and recovery of P from the liquid extract (steps 2 and 3) generated by litter washing in step 1 (fig. 1). Both

experiments were done in the laboratory. The third experiment was a pilot evaluation done in the field with a prototype system at a larger scale to confirm laboratory observations and derive process recommendations.

LABORATORY EXPERIMENTS

Experiment 1

Organic and inorganic acids were tested for their potential to extract P (step 1) from poultry litter using the quick wash process. Aqueous solutions of acetic, citric, and hydrochloric acids were added to 2.00 g poultry litter samples (1:25 w/v ratio) in 50 mL graduated glass test tubes at seven concentration levels (0, 2.5, 5, 10, 20, 40, and 80 mmol L^{-1}). The solutions and litter were mixed in a reciprocating shaker (135 oscillations min^{-1}) at ambient temperature (23°C) for 1 h. Subsequently, solids and liquid were separated by centrifuge (2000g) for 5 min. The liquid supernatant was decanted and analyzed for pH, total P (TP), and total Kjeldahl N (TKN). Solids were dried at 40°C in a forced-air drier and analyzed for TKN and TP. The extraction experiment was conducted in duplicate, and the treatment control consisted of extraction with distilled water. Treatment efficiency of the various acid treatments was established by comparison of P extraction relative to initial P content in untreated poultry litter.

Experiment 2

This experiment was carried out to demonstrate the removal and recovery of P from the liquid extract (steps 2 and 3) generated by litter washing in step 1 (fig. 1). Poultry litter (64 g) was mixed (1:25 w/v) with 1.6 L of a 20 mM citric acid solution in a 2 L beaker and stirred for 1 h with a magnetic stirrer. After the mixture settled for 20 min, the liquid extract was separated from the washed litter by decantation and transferred 35 mL aliquots to separate laboratory vessels (50 mL graduated glass tubes). To one half of the vessels, we applied hydrated lime [$\text{Ca}(\text{OH})_2$] treatments; to the other half, we applied $\text{Ca}(\text{OH})_2$ and flocculant treatment (steps 2 and 3). A 2% $\text{Ca}(\text{OH})_2$ solution in water was added in various amounts until the pH of the mixed liquid reached setpoints of 6, 7, 8, 9, 10, or 11 units (treatments 1 to 6, respectively); a control treatment with no lime addition was included (treatment 0). The recovery of P was enhanced by adding an organic flocculant after reaching the setpoint pH (step 3). The organic flocculant was an anionic polyacrylamide (PAM), Magnafloc 120L with 34% mole charge and 50% active ingredient (Ciba Specialty Chemicals Water Treatment, Inc., Suffolk, Va.). This flocculant was added at a rate of 7.0 mg active ingredient (a.i.) per L and mixed for 30 s. For both lime only and lime plus flocculant addition tests, the liquid supernatant was decanted and analyzed for pH, TP, and TKN. Solids were dried at 40°C in a forced-air drier and analyzed for TKN and TP. Treatment efficiency of the various lime and flocculant treatments was expressed as percentage of P extraction relative to initial P content. All tests were conducted in duplicate at room temperature (23°C) and ambient pressure.

FIELD PROTOTYPE EXPERIMENT

A field prototype system was implemented to evaluate the quick wash process to extract and recover P from poultry litter. The prototype system consisted of two connected reactor vessels (fig. 2). The first vessel in the sequence was

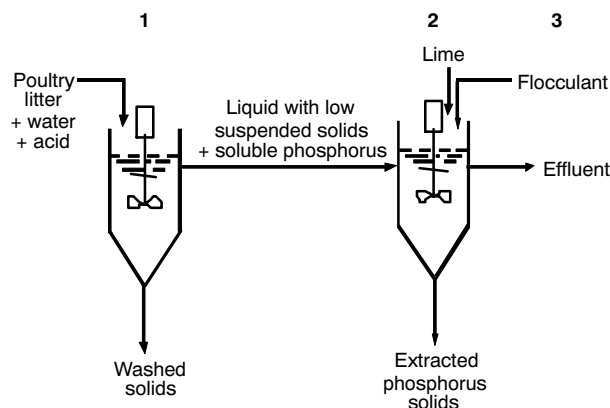


Figure 1. Quick wash process schematic: (1) P extraction, (2) P recovery, and (3) P recovery enhancement.

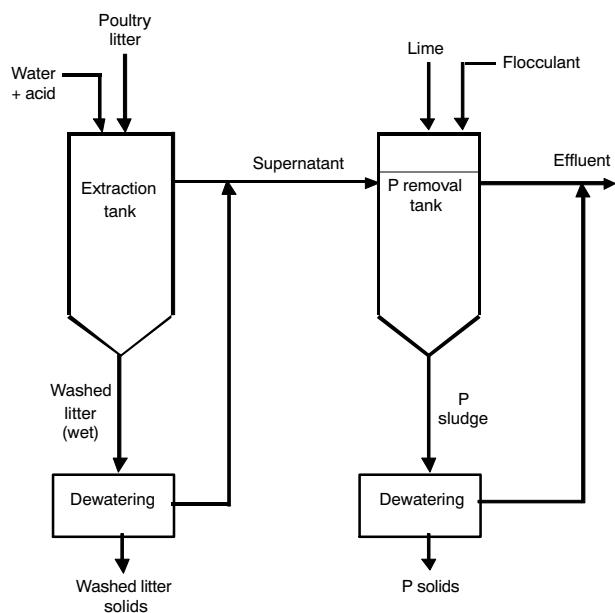


Figure 2. Schematic diagram of the field prototype system for manure quick wash. A mix, reaction, settling, and decantation sequence was used in each tank.

the P extraction reactor, which consisted of a 378 L tank with a conical bottom, a mixer, and a pH controller. Once the liquid reacted with the solids, stirring was stopped to let solids settle. After settling of solids, the supernatant from this tank was pumped to the second vessel. The second vessel in the sequence was the P recovery reactor, which consisted of another 378 L tank with conical bottom, a mixer, and a pH controller. The unit was completed with a smaller 57 L tank with a mixer and pump used to stir and inject the hydrated lime solution into the second vessel.

Each quick wash test was called a “run.” In each run, solids and liquid sampling was done in duplicate. For all runs, P extraction consisted of adding citric acid (10% w/w) to a stirred mixture of broiler litter (15.2 kg) and water (litter-water ratio of 1:25 w/v) inside the extraction reactor. Addition of citric acid stopped when pH of the mixture reached a setpoint of 4.5. The extraction mixture was sampled every 10 min during a 60 min stirring period to determine the minimum stirring time required to reach a stable TP concentration in the extraction liquid; TP was determined in supernatant (after a 24 h settling) of unfiltered samples. The treated litter solids were removed from the bottom of the P extraction reactor after a 20 min settling period and further dewatered through a filter. The filter consisted of a 0.84 × 0.84 × 0.13 m sieve box with a 0.6 cm wire mesh bottom and a 200 µm commercial polypropylene non-woven fabric (E. I. DuPont de Nemours, New Jersey).

The supernatant from the P extraction reactor was pumped into the P recovery reactor tank, where hydrated lime (10% Ca(OH)₂ in water) was injected and mixed. A pH controller stopped lime injection when pH of mixed liquid reached a setpoint of 9.0 (runs 1 and 3) or 10.0 (runs 2 and 4). Once the desired pH was reached, the same flocculant used in experiment 2 (anionic PAM, 34% charge, 50% a.i.) was injected at a rate of 15 mg L⁻¹ (a.i.) and mixed to enhance P recovery for 60 s. The precipitated solids were removed from the bottom of the tank after a 30 min settling period and

Table 1. Broiler litter characteristics.

Experiment	Moisture (%)	TP (g kg ⁻¹)	TN (g kg ⁻¹)	N:P Ratio
Laboratory 1 and 2				
Sample 1	17.6	19.3	34.6	1.8
Sample 2	16.6	19.1	35.5	1.9
Mean (std. error)	17.1 (0.6)	19.2 (0.2)	35.1 (0.6)	1.9 (0.1)
Field Prototype				
Sample 1 (run 1)	29.3	12.8	25.9	2.0
Sample 2 (run 2)	27.9	18.2	26.5	1.5
Sample 3 (run 3)	31.6	14.7	29.4	2.0
Sample 4 (run 4)	20.9	11.6	28.1	2.4
Mean (std. error)	27.4 (2.3)	14.3 (1.4)	27.5 (0.8)	2.0

dewatered through a filter as described above. The dried P solids were analyzed for P, C, N, calcium (Ca), magnesium (Mg), potassium (K), and sodium (Na) content.

POULTRY LITTER CHARACTERISTICS

The bedding material that constituted the base of the broiler litter in all experiments was wood chips. Broiler litter for laboratory experiments 1 and 2 was collected from a 27,400-bird broiler house in Sumter County, South Carolina. At the time of sampling, the litter was being used by the fifth consecutive flock (6.5 flocks per year). Two composite litter samples were taken in two 12 m transects covering the width of the house. Composite samples were placed in 20 L plastic sealed containers and stored in the freezer until preparation for laboratory experiments. Prior to laboratory experiments, litter samples were ground and passed through a 5.8 mm sieve. The broiler litter contained 17.1% (±0.2%) moisture, 19.2 (±0.2) g kg⁻¹ TP, and 35.1 (±0.6) g kg⁻¹ TN (table 1).

Broiler litter used for field prototype experiments was collected from a 25,000-bird broiler house in Lee County, South Carolina. At the time of sampling, the house was empty and between the second and third flock (five flocks per year). Two large composite litter samples were taken in two transects along the house, in its center section (between water lines), and placed in 160 L containers. The containers were sealed, transported, and placed in cold storage (<2°C). Four 15.2 kg samples were prepared for field prototype experiments. Prior to field prototype tests, broiler litter was ground and homogenized using a wood chipper (Yard Machines 5 HP model, MTD, LLC, Cleveland, Ohio). Average particle size distribution of chipped poultry litter is presented in figure 3. On average, the broiler litter contained 27.4% (±2.3%) moisture, 14.3 (±1.4) g kg⁻¹ TP, and 27.5 (±0.8) g kg⁻¹ TN (table 1).

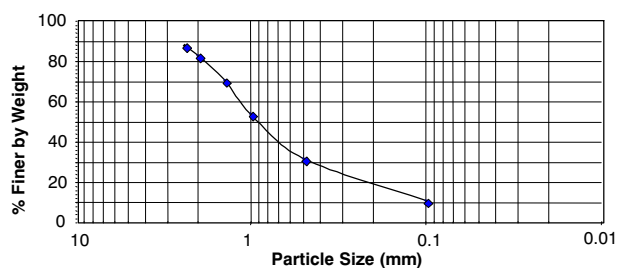


Figure 3. Particle size distribution of homogenized broiler litter used in field prototype experiments. Each data point is the mean of three replicate samples shaken by hand for 1 min to pass through ASTM Standard Sieves Nos. 7, 8, 10, 14, 18, 35, and 140 (Dual Mfg. Co., Chicago, Ill).

ANALYTICAL METHODS

Analyses of supernatant liquid were performed according to *Standard Methods for the Examination of Water and Wastewater* (APHA, 1998). Total P and TKN were determined in liquid and solid samples using the automated ascorbic acid method (Standard Method 4500-P F) and the phenate method (Standard Method 4500-NH₃ G) adapted to digested extracts using H₂SO₄ (Pote and Daniel, 2000), respectively. Total N is the sum of TKN plus nitrate-N. Nitrate-N was also determined using Standard Method 4500-NO₃⁻ F; it represented less than 3% of TN. The pH of the supernatant liquid was measured electrometrically using a combination pH electrode (Standard Method 4500-H⁺ B). Total suspended solids (TSS) were determined by retaining solids on a glass-fiber filter (Whatman grade 934AH, Whatman, Inc., Clifton, N.J.) dried to 105°C (Standard Method 2540 D). Washed litter and P-recovered solids were dried at 40°C in a forced-air drier prior to total C and N analyses. In addition, moisture in recovered solids of the field prototype was determined using a microwave moisture analyzer (Omnimark Instrument Corp., Tempe, Ariz.), which dries solids to a constant weight at 105°C. Elemental analysis of recovered washed and P-rich solids for total C and N was done by dry combustion (Leco Corp., St. Joseph, Mich.); Ca, Mg, K, Na, and P were analyzed by inductively coupled plasma (ICP) from nitric acid plus H₂O₂ digested extract (Peters et al., 2003). Data were statistically analyzed by means and standard errors (proc MEANS), linear regression (PROC REG), analysis of variance (proc ANOVA), and least significant difference at a 0.05 probability level (LSD_{0.05}) for multiple comparisons among means with SAS Version 8 (SAS, 1999).

RESULTS AND DISCUSSION

LABORATORY EXPERIMENTS

Experiment 1

Phosphorus in poultry litter was extracted with increasing molar concentrations (0 to 80 mmol L⁻¹) of both mineral and organic acids (fig. 4). During extraction, a significant portion of total P in poultry litter was released from the litter solids. Total P extraction rates increased with increasing acid concentrations. At 40 mmol L⁻¹ concentration of acid, about 81% of the initial TP content in broiler litter was extracted. In contrast, the distilled water wash (control) extracted only

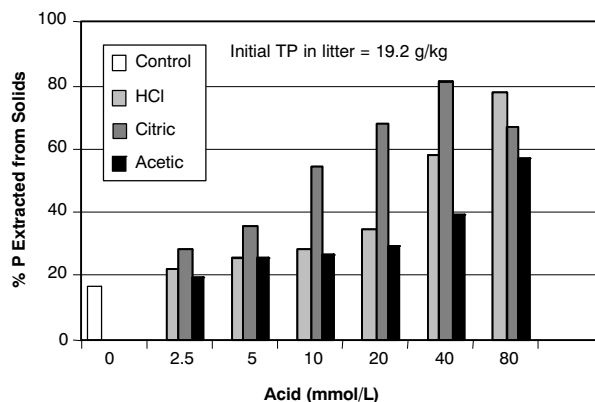


Figure 4. Extraction of phosphorus from poultry litter using acids at seven concentration levels (LSD_{0.05} = 10).

20%. In addition to the concentration of acid, the type of acid made a difference. Citric acid was more efficient at extracting P than HCl or acetic acid at similar molar applications (2.5 to 40 mmol L⁻¹). High extraction efficiencies (>70%) were also possible with HCl and acetic acid (>50%), but the required molar rates were doubled (80 mmol L⁻¹).

Even though P extraction increased from 17% to 81% with increased citric acid treatment in the range of 0 to 40 mmol L⁻¹, N extraction was not greatly affected (table 2). Nitrogen contained in the litter was extracted much less efficiently than P. For instance, about 81% of initial total P in the litter was extracted in treatment 5 at pH 3.8 (40 mM citric acid), but only 27% of N was extracted (table 2). Thus, the litter wash residue resulted in an N:P ratio of 9.8. This is about 5-fold higher than the N:P ratio of the untreated litter (N:P = 2.1). Furthermore, this 9.8 N:P ratio is within the range required for balanced fertilization of crops for both N and P (Edwards and Daniels, 1992).

The percentage of P extracted from solids increased linearly with decreasing pH ($y = -11x + 107$, $R^2 = 0.87$, $n = 19$, $P < 0.0001$; fig. 5). Although the quick wash process consistently extracted more than 50% of TP when pH of the acid solution-broiler litter mixture was lower than 5, similar percentages of P from broiler litter were extracted at different acid concentrations (fig. 5). Thus, the amount of acid added in the process to extract a specific amount of P can be controlled by setting a specific endpoint pH using a pH controller.

Table 2. Effect of citric acid treatment on pH of the extraction solution-solids mixture, total P and N extracted, and N:P ratio in solid residue left after washing poultry litter.

Treatment	pH Mixture ^[a]	Acid (mmol L ⁻¹)	Total N Extracted ^[b]		Total P Remaining in Washed Litter (g kg ⁻¹) ^[a]	Total P Extracted ^[c]		Total P Mass Recovery ^[d] (%)	N:P Ratio Washed Litter ^[e]
			(g kg ⁻¹ litter) ^[a]	(%)		(g kg ⁻¹ litter) ^[a]	(%)		
0	8.2 (0.1)	0.0	10.2 (0.6)	29.1	18.0 (1.9)	3.3 (0.5)	17	111	1.2
1	7.1 (0.1)	2.5	11.6 (0.6)	33.1	16.1 (0.1)	5.5 (0.1)	29	112	1.3
2	6.4 (0.1)	5	11.1 (0.5)	31.7	15.6 (0)	6.9 (0.1)	36	117	1.4
3	5.4 (0.1)	10	11.4 (0.1)	32.5	9.6 (1.2)	11.0 (0.1)	55	107	2.5
4	4.5 (0.1)	20	9.6 (0.3)	27.4	5.1 (0.1)	13.0 (0.7)	68	94	5.5
5	3.8 (0.0)	40	9.4 (0.1)	26.8	3.1 (0.4)	16.0 (0.7)	81	99	9.8
6	3.1 (0.1)	80	7.7 (1.2)	22.0	3.1 (0.1)	13.0 (2.8)	67	84	11.1

^[a] Data are means of two replicates; values in parentheses are standard errors of the mean.

^[b] Total N extracted = TN extraction relative to initial TN content in litter (35.1 g kg⁻¹); LSD_{0.05} = 2.0.

^[c] Total P extracted = P extraction relative to initial P content in litter (19.2 g kg⁻¹); LSD_{0.05} = 4.8.

^[d] Total P mass recovery = ((TP Remaining in Washed Litter + TP extracted)/19.2) × 100.

^[e] Ratio calculated using N and P concentration in digested samples.

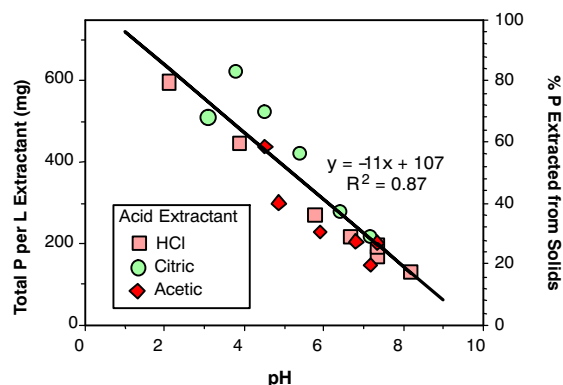


Figure 5. Effect of pH on TP extracted from broiler litter. Total P concentration increased with decreasing pH of mineral and organic acids extracting solutions; more than 50% of TP was extracted with respect to initial TP content in broiler litter at pH of the extracting acid solutions lower than 5. The % P extracted from solids corresponds with values in figure 5. Variables in regression line $y = -11x + 107$ are $x = \text{pH}$ and $y = \% \text{ P extracted from solids}$.

Although other mineral and organic acids can be used for the quick wash process (such as sulfuric, malic, oxalic, phosphoric, nitric, ethyldiamintetracetic) besides the ones presented in figure 4, the preferred acids for quick wash are those that do not add P or N during the quick wash process. However, the use of acids such as nitric, ethyldiamintetracetic, sulfuric, or phosphoric may be useful to fortify the final extracted P product with N, sulfur, or P.

From the results of experiment 1, we concluded that with the quick wash process, the treated litter (washed solids) could be land applied at application rates based on N crop requirements without accumulation of excess P in the soil. For instance, using data from Edwards and Daniel (1992), an N:P ratio of 5.2:1 would be needed to match Kentucky bluegrass specific nutrient uptake needs, which can be delivered with a P extraction at pH 4.5 (N:P = 5.5). Higher N:P ratios needed for cotton (6.2:1), corn (7.5:1), or wheat (10.7:1) can be obtained at $\text{pH} < 4.5$ (table 2).

Experiment 2

A 20 mmol L^{-1} citric acid extract solution was selected for step 1 for further recovery of P with hydrated lime. This liquid extract contained a high TP concentration of about 600 mg L^{-1}

Table 3. Quick wash process (step 2), hydrated lime application for recovery of extracted soluble P from broiler litter. Data show TP concentration in liquid extract and corresponding percentage of TP removed by increasing pH with hydrated lime after P extraction (step 1) with 20 mM citric acid solution (1:25 w/v).

Treatment ^[a]	pH	Ca(OH) ₂ Applied (g L^{-1} liquid)	Total P in Liquid Extract ^[b] (mg L^{-1})	Total P Removed from Liquid Extract ^[c] (%)
0	4.7	0.0	613 (11)	0
1	6.0	1.4	381 (9)	39
2	7.0	2.0	299 (10)	51
3	8.0	2.6	215 (48)	65
4	9.0	3.1	251 (33)	59
5	10.0	3.7	303 (28)	51
6	11.0	4.1	237 (19)	62

^[a] Treatment of the liquid was done by addition of hydrated lime (2% Ca(OH)₂ in water) to obtain a specific pH.

^[b] Data are the means of two replicates; values in parenthesis are standard errors of the mean.

^[c] Total P removed = P recovered from liquid fraction relative to initial P concentration in liquid extract (613 mg L^{-1}).

at pH 4.7 (table 3, treatment 0) and a low TSS concentration (2.1 g L^{-1}) after liquid-solid separation by decantation. In step 2, TP was removed from solution by precipitating soluble P compounds under alkaline conditions. Addition of hydrated lime decreased TP until a pH of 8.0 units was obtained (table 3).

Subsequent addition of a flocculant improved the percentage of TP removed at pH higher than 8.0 (table 4). A small amount of an organic flocculant was added at a rate of 7 mg L^{-1} (active ingredient) to all treatments to enhance thickening and TP content in the precipitated product (step 3). Results in table 4 show an increase of the amount of P extracted and higher TP content of the precipitate by addition of Ca (OH)₂ followed by flocculant enhancement. The highest P extraction rate and content in the precipitate (18.8% P_2O_5) was obtained when pH reached a value of 10.0.

The enhancing effect of organic flocculant addition on TP content of the precipitate is summarized in table 5 at three hydrated lime levels (pH 8, 9, and 10) with and without application of polymer after citric acid (20 mM) extraction. From these results, we concluded that after acid extraction >60% of total P in poultry litter can be recovered by the addition of hydrated lime and small amounts of organic flocculant (steps 2 and 3).

Table 4. Quick wash process (steps 2 and 3), hydrated lime plus flocculant application for recovery of extracted soluble P from broiler litter. Data show total P remaining in liquid effluent, recovered per unit weight of broiler litter, and P grade of the precipitate produced by increasing pH with Ca(OH)₂ and addition of organic flocculant after P extraction (step 1) with 20 mM citric acid solution (1:25 w/v).

Treatment ^[a]	pH	Ca(OH) ₂ Applied (g kg^{-1} litter)	Total P Remaining in Liquid ^[b]	Total P in Precipitate ^[b] (g kg^{-1} litter)	Total P Recovered in Precipitate ^[c] (%)	Total P Mass Recovery ^[d] (%)	P Grade in Precipitate ^[b] (% P_2O_5)
0	4.7	0.0	613 (11)	0.5 (0.1)	2.8	106	1.4 (0.3)
1	6.0	36	355 (4)	6.5 (0.2)	33.6	107	14.9 (7.0)
2	7.0	50	202 (16)	8.1 (0.5)	42.3	95	11.9 (3.3)
3	8.0	65	103 (4)	11.7 (0.1)	61.0	101	17.6 (4.4)
4	9.0	78	94 (17)	13.0 (0.2)	67.5	107	17.2 (0.6)
5	10.0	93	15 (2)	13.9 (0.1)	72.5	99	18.8 (1.2)
6	11.0	104	13 (4)	13.5 (0.0)	70.4	98	14.4 (4.4)

^[a] Treatment of the liquid was done by addition of hydrated lime (2% Ca(OH)₂ in water) to obtain a specific pH. An anionic polymer (polyacrylamide, 34% charge) was added at a rate of 7 mg L^{-1} (active ingredient) to all treatments to enhance precipitation.

^[b] Data are means of two replicates (standard errors in parentheses).

^[c] Total P recovered in precipitate = TP in the precipitated solids relative to initial P content in litter (19.2 g kg^{-1}).

^[d] Total P mass recovery = [(washed litter + precipitate + liquid effluent) / initial P content in litter] $\times 100$; washed litter contained 5.0 g TP kg^{-1} .

Table 5. Total recovered P in precipitate increased by organic flocculant addition (step 3). Data show variable hydrated lime addition without and with anionic polyacrylamide application.

Lime Treatment	pH ^[a]	Total P Recovered in Precipitate (g kg ⁻¹ litter) ^[b]		Recovery Increase with Polymer (%)
		Without Polymer	With Polymer ^[c]	
1	8.0	10.0 (1.2)	11.7 (0.1)	14.0
2	9.0	9.1 (0.8)	13.0 (0.2)	30.0
3	10.0	7.7 (0.7)	13.9 (0.1)	45.0

^[a] Specific pH values obtained using hydrated lime (2% Ca(OH)₂ in water).

^[b] Total P recovered = P removal from liquid fraction relative to initial P content in litter (19.2 g kg⁻¹). Data are means of two replicates (standard errors in parentheses).

^[c] Anionic polyacrylamide, 34% charge, applied at a constant rate (7 mg L⁻¹ active ingredient).

FIELD PROTOTYPE EXPERIMENT

The prototype experiment was based on the acid and alkaline endpoint pH values that were determined in laboratory experiments 1 and 2 to extract and recover >60% of TP from poultry litter. To avoid an excessive chemical application, the prototype experiment extracted P from broiler litter at a solution pH of 4.5 with citric acid. The first tested component was the effect of stirring time on amount of P extracted from the slurry formed by mixing litter and extracting liquid (step 1). Extracted TP concentration remained stable (300 to 330 mg L⁻¹) at pH 4.5 with stirring time of 20 min or more (fig. 6). From these results, we confirmed that stirring time of 20 to 60 min is sufficient to obtain a stable TP extracted concentration during extraction process at pH less than 5.0.

Phosphorus extraction performance of the prototype system under field conditions (table 6) was consistent with performance obtained in the laboratory (figs. 4 and 5). On average, the N:P ratio of the broiler litter increased from 2.0 before wash treatment to 4.6 after washing (table 6). This higher N:P ratio is better for crop utilization. As an alternative, the washed litter could be reused in the broiler house as bedding materials or used for on-farm bioenergy production. Phosphorus extraction efficiencies of 65% to 89% with respect to initial TP in broiler litter were obtained with pH treatment of 4.5 for all four runs of the pilot tests (table 6).

The supernatant liquid in the P extraction tank (fig. 2) had a low TSS concentration (<3.5 g L⁻¹) with respect to the TSS concentration of the extraction slurry (>28.7 g L⁻¹) in all four runs after solids settling. Thus, this clarified liquid was pumped to the P recovery tank and reacted with hydrated lime and flocculant. The complete process recovered >60% of the

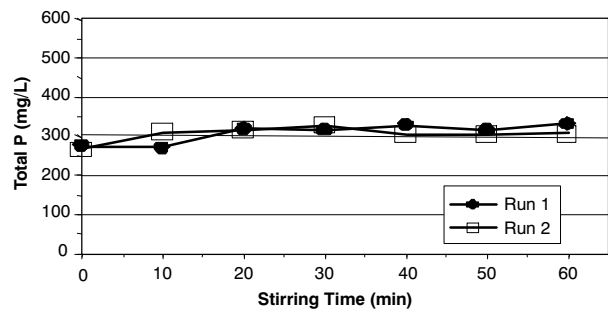


Figure 6. Effect of stirring time on total P concentration in the extract. Broiler litter was extracted with citric acid solution at pH 4.5 (step 1). Data show that TP concentration stays stable in supernatant liquid with stirring time between 20 and 60 min. Data points are average concentrations of two separate runs using the field prototype.

initial total P in broiler litter; these high P recovery rates were obtained both at extraction pH of 9.0 and 10.0 units (table 6).

Before dewatering, mean initial moisture of the recovered P sludge was about 95.5% (table 7). After filtration, the P sludge had a mean moisture content of 89.8%. The drying process was further accelerated by placing the recovered P sludge to dry in a greenhouse. The mean moisture content declined to <10% in the subsequent 13 days after filtration (table 7).

The prototype performance confirmed laboratory results that >60% of the total P content of poultry litter can be recovered using the quick wash process (table 6). The P grade of the product recovered in the four prototype runs was lower on average (13.5% P₂O₅ = 5.9 g P/100 g × 2.29) than the precipitate obtained in the laboratory at pH 9 and 10 (17.2% and 18.8% P₂O₅, respectively, table 4). These differences are most probably due to differences in initial moisture content of the broiler litter samples. For example, on a dry matter basis, litter treated in the prototype experiment had a lower mass and lower P concentration per volume of extracting solution, which certainly affected the P grade of the precipitated product. In the prototype experiment, litter samples had an average lower TP content (14.3 mg kg⁻¹) and much higher moisture (27.4%) than the broiler litter used in laboratory studies (TP 19.2 mg kg⁻¹ and 17.1% moisture; table 1). Thus, to obtain a high P grade material, both the moisture and P content of poultry litter should be determined for each batch in order to adjust the weight of fresh litter treated per volume of extracting solution.

On average, the recovered precipitate contained relatively large amounts of P, C, N, and Ca, and small amounts of Mg, K, and Na (table 8). These results confirm our laboratory data that the quick wash approach can extract and recover P from

Table 6. Mass balance and performance of field prototype to extract and recover phosphorus from poultry litter using the quick wash process.

Run	Litter Before Wash		N:P Ratio Washed Litter	pH	Extraction		pH	Recovery	
	Total P (g kg ⁻¹ litter)	N:P Ratio ^[a]			Total P ^[b]			Total P ^[c]	
					(g kg ⁻¹ litter)	(%)		(g kg ⁻¹ litter)	(%)
1	12.8	2.0	4.4	4.5	8.3	65	9.0	7.7	60
2	18.2	1.5	4.1	4.5	13.7	75	10.0	12.2	67
3	14.7	2.0	5.8	4.5	13.1	89	9.0	12.0	82
4	11.6	2.4	4.0	4.5	9.4	81	10.0	7.3	63
Average	14.3 (1.4)	2.0 (0.2)	4.6 (0.4)		11.1 (1.3)	78 (5)		9.8 (1.3)	68 (5)

^[a] Initial N content in litter: 25.9, 26.5, 29.4, and 28.1 g kg⁻¹ for runs 1, 2, 3, and 4, respectively.

^[b] Total P extracted = P extracted relative to initial P content in litter before wash.

^[c] Total P recovered = P recovered in precipitated solids relative to initial P content in litter after flocculant application.

Table 7. Percent moisture of recovered phosphorus sludge before and after drying in greenhouse.

Sludge P Dewatering	Percent Moisture (g/100 g)				
	Run 1	Run 2	Run 3	Run 4	Mean ^[a]
Initial moisture ^[b]	96.0	96.5	94.8	94.7	95.5 (0.5)
After filtering ^[c]	89.0	88.6	89.5	92.0	89.8 (0.8)
Air dried ^[d]	10.1	9.1	10.4	10.4	9.9 (0.4)

[a] Mean (standard error of the mean).

[b] Sludge obtained by decantation of liquid after flocculant addition (step 3).

[c] Dewatering for 24 h after filtration through polypropylene non-woven filter fabric.

[d] Air dried for 13 days after dewatering in greenhouse; average temperature = 37°C and relative humidity = 54%.

poultry litter in a concentrated product that has the potential of being reused as fertilizer. An additional characteristic of the recovered P product was its reduced bulk volume. The air-dried recovered P product (average dry bulk density of 780 g dm⁻³) had about 17% of the initial volume of poultry litter. Therefore, the recovered P product can be transported more economically off the farm for use as a fertilizer material.

ECONOMIC CONSIDERATIONS

The chemical cost to extract and precipitate P from poultry litter using the quick wash process was calculated on the basis of treating one metric ton of poultry litter. Treatment parameters and target values (table 9) used in these calculations are based on the average performance of our field prototype experiment with the following conditions:

- The washed litter and effluents are land applied on N basis.
- Poultry litter contains 20 g TP kg⁻¹ and 35 g TN kg⁻¹.
- The efficiency for TP recovery in the precipitate is 70%.
- Sulfuric acid (H₂SO₄) is used in step 1 to remove P from litter.

We assumed that the quick wash treatment is done on-farm to eliminate the cost of transporting untreated poultry litter to a centralized facility. The washed litter and effluent remain on-farm and are reused as crop fertilizer. The chemical cost includes the cost of acid, Ca(OH)₂ and flocculant used in the process. We propose to use H₂SO₄ because it may be more

Table 8. Selected element composition in percent of the weight of the solid precipitate recovered from poultry litter using the quick wash process.^[a]

Constituent	Percent Composition (g/100 g) ^[b]				
	Run 1	Run 2	Run 3	Run 4	Mean ^[c]
Phosphorus	4.6	4.8	8.9	5.3	5.9 (1.0)
P ₂ O ₅ ^[d]	10.5	11.0	20.4	12.1	13.5 (2.3)
Carbon	35.6	36.1	16.1	7.2	23.8 (7.2)
Nitrogen	3.6	3.5	1.8	0.8	2.4 (0.7)
Calcium	11.9	10.5	20.0	11.3	13.4 (2.2)
Magnesium	0.7	0.7	1.5	0.9	1.0 (0.4)
Potassium	0.9	1.0	1.8	0.6	1.1 (0.3)
Sodium	0.3	0.3	0.5	0.2	0.3 (0.1)

[a] Data for runs 1 and 3 were obtained at pH 9; data for runs 2 and 4 were obtained at pH 10 (table 6).

[b] Percent composition expressed as oven dry values.

[c] Mean (standard error of the mean).

[d] Phosphorus grade expressed as P₂O₅ = % P × 2.29.

Table 9. Volume and nutrient content of washed litter; P sludge, and effluent produced per metric ton of poultry litter treated using the quick wash system.

Parameter	Unit	Extraction (step 1)		Recovery (steps 2 and 3)	
		Washed Litter ^[a]	Liquid	P Sludge	Effluent
Volume ^[b]	m ³	2.0	23	3	21
TP	kg	3	--	14	1
TN	kg	24	--	5	6

[a] Initial poultry litter content of 20 g TP kg⁻¹ and 30 g TN kg⁻¹.

[b] Volume data were calculated from average usage of four field prototype experimental runs (table 6).

economical than other acids. The cost of H₂SO₄ (95%) is about \$329 per metric ton of acid. The P extraction of the quick wash process (step 1) would use 100 kg H₂SO₄ at a cost of \$32.9 per ton of washed litter. The cost of prepared hydrated lime slurry (30% hydrated lime, 3.01 lb gal⁻¹) in tote tanks is approximately \$0.204 kg⁻¹ of dry hydrated lime. In step 2, the cost of 47 kg of Ca(OH)₂ used per ton of treated litter is \$9.59. The cost of polymer is about \$3.96 kg⁻¹ (\$1.80 lb⁻¹). The P precipitation enhancement (step 3) requires about 0.4 kg of anionic PAM (50%) with a cost of \$1.58 per metric ton of treated litter. This results in a total chemical cost of \$44.07 per ton of poultry litter.

The amount of P potentially recovered in the solids produced using the quick wash approach that can be sold is about 14 kg P or 32 kg P₂O₅ per metric ton of poultry litter with a fertilizer value of \$63.36 (\$0.90 lb⁻¹ P₂O₅ or \$1.98 kg⁻¹ P₂O₅). It is anticipated that water quality credits will be an important benefit to farmers adopting new manure treatment technologies (EPA, 2004; Ribaud et al., 2007). For current credit prices of \$11.11 kg⁻¹ of P (Chesapeake Bay Watershed Nutrient Credit Exchange, 2007) and trading ratios for nonpoint sources of 2:1, the potential benefit from removing 14 kg P per ton of poultry litter is \$77.8. As an additional benefit, a total of about 30 kg N (\$0.87 kg⁻¹ N) and 4 kg P remain on-farm (washed litter plus effluent) that can be used more effectively.

A complete economic analysis of the technology for on-farm recovery of P would need to consider annualized capital, energy, and labor costs as well as other benefits such as reduction of land area required to dispose manure P.

CONCLUSIONS

We found that >60% of initial total P can be extracted and recovered from poultry litter solids using the quick wash treatment process. Our findings indicate that by washing the poultry litter with a solution of organic or mineral acids, insoluble P is converted to soluble P by rapid hydrolysis reactions. More than 60% of TP with respect to initial TP of the solid manure was extracted when acid extraction slurry had a pH < 5.0. The washed residual litter solid was separated from the liquid extract to prevent unnecessary C and organic N oxidation and digestion. In practice, the acid is added in the minimum amount needed to balance the N:P ratio of the washed residue to produce a residue that is more environmentally safe for land application and use by crops. Once the liquid extract is separated from the solid residue, P is precipitated by addition of lime to the liquid extract to form an alkaline earth metal-containing P compound. Sub-

sequently, an organic poly-electrolyte is added to enhance precipitation and grade of the P product. This quick wash approach extracts and recovers P from poultry litter, producing a concentrated P product that can be moved more easily off-farm and reused as fertilizer. A preliminary chemical cost analysis indicates that the quick wash technology for recovery of P from poultry litter could be cost-effective. Yet, the economic analysis should further consider annualized capital, energy, and labor costs as well as benefits such as farm revenues from exporting the P product for fertilizer use, trading of nutrient credits, and reduction of land area needed for disposal of manure P.

ACKNOWLEDGEMENTS

This research was part of USDA-ARS National Program 206: Manure and By-product Utilization; ARS Project 6657-13630-003-00D "Innovative Animal Manure Treatment Technologies for Enhanced Environmental Quality."

REFERENCES

- APHA. 1998. *Standard Methods for the Examination of Water and Wastewater*. 20th ed. Washington, D.C.: American Public Health Association.
- Chesapeake Bay Watershed Nutrient Credit Exchange. 2007. Status of water quality credit trading in Virginia. Richmond, Va.: Department of Environmental Quality. Available at: http://agenpolicy.aers.psu.edu/Trading/VA_Trading_Sum_MidAtlanticExt%202_Mar_07.doc.
- Edwards, D. R., and T. C. Daniel. 1992. Environmental impacts of on-farm poultry waste disposal: A review. *Bioresour. Tech.* 41(1): 9-33.
- EPA. 2004. Water quality trading assessment handbook. Publication EPA 841-B-04-001. Washington, D.C.: U.S. Environmental Protection Agency.
- Heathwaite, L., A. Sharpley, and W. Gburek. 2000. A conceptual approach for integrating phosphorus and nitrogen management at watershed scales. *J. Environ. Qual.* 29(1): 158-166.
- Jones, K., and G. D'Souza. 2001. Trading poultry litter at the watershed level: A goal focusing application. *Agric. Resour. Econ. Rev.* 30(1): 56-65.
- Kelleher, B. P., J. J. Leahy, A. M. Henihan, T. F. O'Dwyer, D. Sutton, and M. J. Leahy. 2002. Advances in poultry litter disposal technology: A review. *Bioresour. Tech.* 83(1): 27-36.
- Kellogg, R. L., C. H. Lander, D. C. Moffitt, and N. Gollehon. 2000. Manure nutrients relative to the capacity of cropland and pastureland to assimilate nutrients: Spatial and temporal trends for the United States. GSA Publ. No. nps00-0579. Washington, D.C.: USDA-NRCS and USDA-ERS.
- Keplinger, K. O., and L. M. Hauck. 2004. Impacts of livestock concentration and application rate restrictions on manure utilization. ASAE/CSAE Paper No. 042204. St. Joseph, Mich.: ASAE.
- Moore, P. A. 2002. Best management practices for poultry manure utilization that enhance agricultural productivity and reduce pollution. In *Animal Waste Utilization: Effective Use of Manure as a Soil Resource*, 89-123. J. L. Hatfield and B. A. Stewart, eds. Boca Raton, Fla.: Lewis Publishers.
- Peters, J., S. M. Combs, B. Hoskins, J. Jarman, J. L. Kovar, M. E. Watson, A. M. Wolf, and N. Wolf. 2003. Recommended methods of manure analysis. Publication A3769. Madison, Wisc.: University of Wisconsin Extension.
- Potash and Phosphate Institute. 2002. Plant nutrient use in North American agriculture. Technical Bulletin 2002-1. Norcross, Ga.: Potash and Phosphate Institute.
- Pote, D. H., and T. C. Daniel. 2000. Analyzing for total phosphorus and total dissolved phosphorus in water samples. In *Methods of Phosphorus Analysis for Soils, Sediments, Residuals, and Water*, 94-97. G. M. Pierzynski, ed. So. Coop. Series Bull. No. 396. Manhattan, Kans.: Kansas State University.
- Ribaudo, M., R. Johansson, and C. Jones. 2007. Environmental credit trading: Can farming benefit? *Amber Waves* 26(1): 48-51.
- SAS. 1999. SAS Version 8.00. Cary, N.C.: SAS Institute, Inc.
- Sharpley, A., B. Foy, and P. Withers. 2000. Practical and innovative measures for the control of agricultural phosphorus losses to water: An overview. *J. Environ. Qual.* 29(1): 1-9.
- Sharpley, A. N., S. Herron, and T. Daniel. 2007. Overcoming the challenges of phosphorus-based management challenges in poultry farming. *J. Soil Water Cons.* 62(6): 375-389.
- Sheth, A. C., and A. D. Turner. 2002. Kinetics and economics of catalytic steam gasification of broiler litter. *Trans. ASAE* 45(4): 1111-1121.
- Smil, V. 2000. Phosphorus in the environment: Natural flows and human interferences. *Ann. Rev. Energy Environ.* 25(1): 53-88.
- Szögi, A. A., M. B. Vanotti, and P. G. Hunt. 2008. Process for removing and recovering phosphorus from animal waste. Patent Application Serial No. 12/026/246. Washington, D.C.: U.S. Patent and Trademark Office.
- USDA-ERS. 2000. Confined animal production poses manure management problems. *Agric. Outlook* (Sept.): 12-18.
- USDOE. 2000. Biomass co-firing: A renewable alternative for utilities. NREL/FS-570-28009; DOE/GO-102000-1055. Golden, Colo.: U.S. Department of Energy, National Renewable Energy Laboratory.